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STUDIES ON THERMOLUMINESCENCE, METAMICTIZATION AND SINTERING PROPERTIES OF ZIRCON SANDS

A. P. NEURUKAR,† REENA DE, P. N. CHAKRABORTY and I. K. KAUL

Indian Institute of Technology, Kharagpur, India

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Intrinsic properties of zircon sands, varying in colour from pink to yellow and brown, and light green to green and dark green have been studied. This has been done by investigating their present alpha activity, thermoluminescence characteristics, X-ray diffraction patterns, DTA curves and sintering temperatures.

It is found that sintering temperatures are a function of alpha activity. Zircons with dark colours are found to have higher alpha activity and show higher crystalline disorders and lower sintering temperatures and may be unsuited for industrial purposes.

Our TL studies confirm the findings of Vaz and Senftle (1971) and further studies on geochronology of these samples are under investigation.

INTRODUCTION

With increasing demand for quality castings which require better strength properties, more accurate dimensional control and high degree of surface finish, various sands have been tried as a refractory medium for foundry use. Special properties of zircon viz., high conductivity, high density, low thermal expansion and chemical stability make it highly desirable as a moulding medium. In recent times other mineral sands viz., chromite, olivine etc., are being increasingly sought for high temperature casting moulds. The change over to new sands is being made even by some normally zircon sand practitioners due to the fact that zircon sands have failed to yield a substantially improved casting quality over silica sand so much so that in some exceptional cases it has even been poorer to silica sand.

It has long been recognized that zircons show considerable variation in physical properties even though there is little difference in chemical composition. Mineralogists, earlier, classified zircons into three groups on the basis of their optical properties, density and response to heat treatment. Subsequently, in addition to these properties a further criteria, based on chemical alteration by hydration, was utilized for distinction. Further work showed that grouping of zircons according to these properties could not be justified since samples with intermediate properties were also common. It was suggested that a genetic relationship exists between the three groups of constant composition and the differences in their physical properties was attributed to the effects of alpha-particle radiation emanating from the decay products of uranium and thorium present within the crystal structure. It is now well established that the change in density, refractive index, etc., are due to the breakdown of the crystal structure. It has also been shown that for a set of zircons of the same geological age, those containing high concentration of uranium and thorium exhibit higher structural disorders than those containing less amounts (Holland and Gothfried, 1935).

It is generally believed that nuclear radiation is the cause of the observed changes in the physical properties of certain minerals and the phenomenon has been termed metamictization. The term metamict is generally used in mineralogy to denote minerals initially crystalline, but amorphous later, losing their crystal structure in this process of change due to molecular rearrangement caused by

† Present address: Department of Applied Physics, Faculty of Engg. and Technology, M.S. University, Baroda, India.
radioactive emanations from uranium or thorium contained in the mineral. Metamictization occurs in structures which are originally weakly bonded. Even though they may have crystal faces, metamict minerals are generally amorphous to X-rays or yield a faint fuzzy pattern. Some metamict minerals like zircon give sharp electron diffraction patterns owing to retention of relict crystallinity sufficient to permit the relatively short wave electrons to yield resolved patterns (Heinrich, 1958). The mineral gets converted to an isotropic form, with a decrease in specific gravity. Cleavage is absent and fractures are conchoidal. The mineral may be focus of fractures radiating into surrounding mineral grains. There are abnormal amounts of non essential water and a decreased resistance to chemical attack. The mineral may be pyrognomic - recrystallization occurs upon heating, usually at a definite temperature, with a considerable minor evolution of heat and light to an aggregate crystalline pseudomorph, i.e., thermoluminescent.

The disorganization of the crystalline structure may be measured by several methods - TL techniques, density measurements, X-ray diffraction, DTA, etc. Kulp et al. (1952) have shown that the characteristic thermal curve for metamict zircon appears to consist of a doublet at a temperature of 890°C to 910°C, suggesting recrystallization of mineral to be a two stage process. According to Frondel (1953) the differential thermal curve shows a gradual water loss at 100°C to 225°C and a broad exothermic peak at 460°C to 550°C, followed by a single sharp peak at 880°C for reorganization of the metamict material. According to Holland and Gottfried (1935), for zircon completely undamaged by alpha radiation, the X-ray diffraction 28 angle (Cu Ka) for 112 plane is 35.63° while in metamict zircon it approaches to 35.1°. The lattice expansion in metamict zircon is anisotropic with the ratio between the two “a” dimensions, a1/a2 being 1.001 to 0.9735 while the “c” dimension also expands from 5.974°A for crystalline material to 6.080°A for metamict material (Zircon belonging to the tetragonal system). Berman (1955) has described transitions from zircon crystals appearing entirely “fresh” with sharp X-ray diffraction patterns to those that appeared completely metamict and gave no diffraction pattern. In the specimens that were somewhat metamict both the index of refraction and birefringence decreased: complete isotropy was reached in the totally metamict specimen.

Pellas (1965) has reviewed most of the recent measurements, and presented a modern comprehensive model of the metamictization process in zircon, further modification to which has been suggested by Vaz and Senftle (1971). When zircon is subjected to ionizing radiation, viz., alpha, beta and gamma radiation, electrons and holes are produced in the crystal lattice. On heating such a zircon, the trapped charge acquires sufficient thermal energy to escape from its trap, such that it can move in the conduction or the valence band and recombine with the opposite charge. The emission of light during this recombination process provides the source of thermoluminescence. Natural charge trapping in zircons is the result of ionization by the radiation from the decay of radioactive inclusions in the crystal. According to Pellas (1965) the crystal structure of zircon is broken down into four phases during the process of metamictization. Initially the radiation from the radioactive decay of the radioactive inclusions produces an expanded zircon phase that contains a high concentration of interstitials and vacancies. Further radiation damage finally results into a break-up of the ZrSiO4 lattice into ZrO2, SiO2, and amorphous zircon. He further suggests that the concentration of the expanded zircon phase and the three final phases reach maximum concentration at different total alpha particle doses. In view of this model it is expected that the largest number of electron or hole traps would be associated with the phase containing the largest number of interstitials and vacancies. Hence, thermoluminescence of zircon should be maximum at an alpha dose at which the expanded zircon phase reaches a maximum. Krasnobayev (1964) found a sharp drop in natural thermoluminescence of zircons for alpha doses a hundred times less than that expected from Pellas’ model. The present investigation was undertaken on the lines of studies made by Vaz and Senftle (1971) to gather further knowledge on damage mechanism of zircons.

**EXPERIMENTAL METHOD**

For the present study seven zircon samples, varying in colour from pink to brown, light yellow to yellow and light green to green and dark green, were chosen. Further, for comparison, in some parts of this investigation, three known metamict varieties of zircon, viz., Naegite, Alvite and Crytolite were used.
Alpha Activity, Density and Thermoluminescence Measurements

The values of density for different zircon samples used was determined by using two quartz pycnometers of 50 ml capacity. Redistilled water free from air bubbles was used and precautions were taken to reduce the evaporation to a minimum. Air bubbles adhering to the samples were completely removed by heating the pycnometer, containing the sample and 3/4 of its volume of water, to about 50°C under diminished pressure. Weighing was done with the help of an electrical balance, (E. Mettler, Zurich, Switzerland) which measures correctly up to four places of decimal. Corrections were made for the differences in the displacement of air by the volume of the contents of the pycnometers. Three different determinations were made on different portions of each sample and the difference did not exceed 0.0009. The results tabulated (Table I) may be considered correct to the nearest unit in the third decimal place.

Alpha activity was measured by using an alpha probe - SP647A manufactured by electronics corporation of India, utilizing a scintillation detector. Counting time was chosen so as to reduce the counting error to less than three percent. Frequent checks were made to look for any radon loss in the samples and usual corrections for geometry, self absorption and background were taken care of.

Thermoluminescence of the samples was recorded with the experimental arrangement similar to that described by Kaul et al. (1976). This set-up uses the output of an additional PM through a difference amplifier for automatic correction of the black body radiation and natural thermoluminescence if desired. To avoid variation in TL-glow due to grain size, samples with grain size 80–100 mesh were thoroughly mixed and uniformly spread on the TL-oven. The heating rate for glow measurements was kept constant at 20°C/min. linearity being provided by a programmer. The linearity and reproducibility were found to be correct within three to five percent for all TL-glow measurements. Glow curves were recorded under the following conditions:

a) Natural TL measured for each sample using a bleached portion of the sample as a reference for correction of black body radiation.

b) Different aliquots of the same sample were irradiated by different doses of gamma-rays from Co^{60} source and induced TL measured. Correction for natural TL and black body radiation was made by using an unirradiated portion of the particular sample as a reference.

c) The samples were annealed at a constant temperature of 950°C for different periods of time, then quenched to the room temperature and TL measured after irradiation dose of $2.4 \times 10^8$ rads from Co^{60} source. Bleached but unirradiated portion of the sample being used as a reference for correction of black body radiation.

Table 1 shows the measurements on zircon samples.

### Table 1: Measurements on zircon samples

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>Colour</th>
<th>Density gm/cc</th>
<th>Alpha activity alpha/mg-hr</th>
<th>TL ratio</th>
<th>Sintering point °C</th>
<th>2P</th>
</tr>
</thead>
<tbody>
<tr>
<td>B-13</td>
<td>pink</td>
<td>4.69</td>
<td>25</td>
<td>11.50</td>
<td>1.350</td>
<td>35.65</td>
</tr>
<tr>
<td>A-15</td>
<td>light</td>
<td>4.78</td>
<td>63</td>
<td>24.00</td>
<td>1.350</td>
<td>35.64</td>
</tr>
<tr>
<td>C-11</td>
<td>brownish</td>
<td>4.73</td>
<td>91</td>
<td>253.50</td>
<td>1.300</td>
<td>35.62</td>
</tr>
<tr>
<td>D-10</td>
<td>brown</td>
<td>4.64</td>
<td>325</td>
<td>204.50</td>
<td>1.275</td>
<td>35.60</td>
</tr>
<tr>
<td>E-35</td>
<td>green</td>
<td>4.55</td>
<td>540</td>
<td>130.00</td>
<td>1.275</td>
<td>35.60</td>
</tr>
<tr>
<td>F-12</td>
<td>green</td>
<td>4.19</td>
<td>900</td>
<td>43.50</td>
<td>1.250</td>
<td>35.58</td>
</tr>
<tr>
<td>F-15</td>
<td>dark</td>
<td>4.01</td>
<td>2000</td>
<td>1.50</td>
<td>1.245</td>
<td>35.56</td>
</tr>
<tr>
<td>Alvite</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>no pattern</td>
</tr>
<tr>
<td>Naegirte</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>no pattern</td>
</tr>
<tr>
<td>Cyrotlite</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>no pattern</td>
</tr>
</tbody>
</table>

°Ca Kα Ni filter, 30 Kv, 24 MA, 29° drive 1/47 min.
induced TL measured. Correction for natural TL and black body radiation was made by using an unirradiated portion of the sample as reference.

e) The samples were annealed at 950°C for 30 hrs, then quenched to the room temperature and TL measured after irradiation dose of $2.4 \times 10^8$ rads from Co$^{60}$ source. Annealed but unirradiated portion of the sample being used as a reference for the correction of black body radiation.

**Refractoriness or Sintering, DTA and X-Ray Diffraction Measurements**

Refractoriness is an index to the heat resisting properties of a material. This can be quantitatively indicated by criteria like melting point and fusion point of the material, which in general can be considered as any type of phase change. The melting point of a material is the lowest temperature at which the complete conversion from the solid to the liquid state can occur, whereas the fusion point usually refers to the lowest temperature at which any sign of fusion are visible. The deformation of a test piece with temperature - often termed the softening point is at times substituted to indicate the refractoriness as in “Seger cone.” There is no simple, direct relationship between the softening point and the true melting point of a ceramic material. The true melting point is difficult to be conveniently determined but the softening point serves as an indication of the commencement of a phase change. The term “sintering” or “sintering point” has also been used by different workers to indicate a measure of refractoriness of the material.

When a packed granular material is heated, neglecting impurities, liquid phases are first formed out of the smaller particles on the surface and these occupy the interstitials between the solid particles. This process begins at a temperature generally far below the melting point of the material. Initially on the surface there appear independent spots of fused material, but as heating continues further, these spots merge into one another. So long as the fusion is superficial while the bulk of the packed material remains porous, the term sintering is used, but when the fused matter fills the interstitials the mass is said to be vitrified. In ceramics, the refractoriness is commonly measured by pyrometers and thermoscopes, of which Seger cones are very widely used.

An applicable to foundry sands, according to the AFS (1963), sintering point is the temperature level at which incipient fusion has occurred when the test specimen is subjected to controlled conditions of time and temperature. As applied to castings, “sintering point” is that temperature at which the moulding material begins to adhere to the castings, and in sand it is that temperature at which the sand coheres to a platinum ribbon under controlled conditions (Osborn, 1967). The AFS method on sintering point determination is, however, a “non standard” test. The accepted specifications of zircon sands for foundry applications have been proposed by the Steel Founders' Society of America (Middleton, 1969), which stipulates apart from other requirements that the sintering and melting points are to be 2315°C and 2432°C respectively.

For a binary system as is the case of zircon, the solidus point is an appropriate indication of the commencement of fusion. This, of course, does not take the effect of impurities into consideration. There are two main versions of the ZrO$_2$-SiO$_2$ phase diagram (Wilhalm, 1965). The diagram given by Geller and Lang (1959, quoted-Wilhalm, 1965), shown in Figure 1, and that by Curtis and Sowman (1953, quoted-Wilhalm, 1965), given in Figure 2 show a discrepancy in the position of the

![FIGURE 1 System SiO$_2$-ZrO$_2$ (Geller and Lang) phase diagram 132, National Bureau of Standards (U.S.A.)](image-url)
vertical line for the compound zircon $\text{ZrO}_2-\text{SiO}_2$. The diagram of the system $\text{SiO}_2-\text{ZrO}_2$, as given by Toropov and Galakhore (1956, quoted-Wilhalm, 1965) agrees with that of Curtis and Sowman for the region of interest. In the diagram of Geller and Lang (Figure 1), two temperatures are fixed - the eutectic temperature of 1675°C. The diagram of Curtis and Sowman (Figure 2) shows zircon dissociating into zirconia and silica at about 1540°C, before melting commences at the eutectic temperature of 1675°C. This diagram indicates thermal dissociation of zircon in the solid state.

The dissociation of zircon is technologically important because the zirconia product exhibits polymorphism so that if the oxide were retained at room temperature, the normally high resistance to thermal shock of zircon arising from its low coefficient of thermal expansion would be destroyed (Ford, 1967). Thus it is clearly seen that even in the ideal condition of purity of zircon, a phase change could occur even at as low a temperature as 1540°C. The decomposition of $\text{ZrSiO}_4$ was established by Cocco and Schromek in 1957 at 1720°C ± 20°C (quoted-Wilhalm, 1965).

The sintering point of the zircon samples was determined by taking a small amount in a porcelain boat and introduced into a tubular furnace. The temperature of the tubular furnace was gradually raised. Beyond 1000°C, the boat was withdrawn for every 25°C rise of temperature, and the sample scrutinised through a magnifying glass. The temperature at which the first indications of fusion were noticed was taken as the sintering point of the sample. In the present studies the maximum temperature attainable in the furnace used was 1350°C. Investigations for X-ray diffraction studies and Differential Thermal Analyses of the said zircon samples were carried out with a view to determine the extent of metamasitization on the lines suggested by Holland and Gottfried (1935) and Kulp et al. (1952). The results of sintering point and X-ray diffraction studies are presented in Table I and the DTA results shown in Figure 3. The micrographs of polished sections of the zircon samples are presented in plates 1 to 5 and the scanning electron micrographs of the aggregate samples shown in plates 6 to 10.

RESULTS AND DISCUSSION

Results of various measurement are presented in an abstract form in Table I. The colour of zircon samples can be correlated to their present alpha activity; the pink and yellow zircons have relatively low alpha activities while deep green zircons have very high alpha activities. The density of the samples decreases with alpha activity following a sigmoid shaped curve. Holland and Gottfried (1955) showed for gem quality zircons from Ceylon a similar relationship with the alpha dose.

No natural thermoluminescence could be observed in green and dark green zircons while other samples showed a broad peak around 235°C, and gamma ray irradiation of the samples induced another peak around 130°C (Figures 4 and 5). The shape of the glow curves indicate the presence of many other peaks, but in the present studies digitalization of the whole emission was taken as a measure of TL. The results of investigation (b) show the variation of the TL emission with increasing gamma-ray dose. In this investigation different aliquots of the same zircon sample were used because zircon is found to be sensitive to preheating (Figure 6). In all the zircon samples, after a gamma ray dose of $2.4 \times 10^8\text{rads}$, the TL emission reaches saturation, the magnitude of which seems to depend on the present alpha activity, i.e., the natural radiation damage suffered by the samples. Because of this a gamma ray dose of $2.4 \times 10^8\text{rads}$ was used as a standard dose in all subsequent TL investigations and on the basis of studies made by Kulp et al. (1952), the temperature of 950°C, at
which the recrystallization of metamict zircon takes place, was chosen for thermal annealing of the samples. It was observed that the thermal treatment removed the colouration of most of the samples.

The results of investigation (c) viz., the effect of annealing time on TL emission for various zircon samples show that the magnitude of change in TL emission and its intensity depend on annealing time as well as the present alpha activity of the samples. The TL emission of the samples with alpha activity greater than 400 alphas/mg-hr show slight change with heating time while the ones with alpha activity less than 300 alphas/mg-hr show a significant change (Figure 6). It is also seen that, after 24 hours of heating at 950°C, the TL emission of all the samples remains relatively unchanged. Due to this fact 30 hours of annealing time at 950°C was chosen as a standard annealing period for the subsequent investigations.

The induced TL emission of the unheated zircon samples reduces by a factor of about $10^5$ for the zircon sample with the present alpha activity of nearly 400 alphas/mg-hr. With increasing alpha activity the TL emission remains practically unchanged. The induced TL emission of annealed
samples substantially increases, and this increase seems to be clearly a function of alpha activity of the zircon samples. The increase in TL emission could be more clearly indicated by plotting the TL ratio after and before annealing against the present activity of the samples (Figure 7). The maximum TL ratio ($TL_{\text{maximum}}$) occurs for the present alpha activity of about 100 alphas/mg-hr and approaches nearly unity around 2000 alphas/mg-hr.
Following the lines of Vaz and Senftle (1970), if it is assumed that the TL emission before heating is contributed by undamaged zircon (Z₁), whereas the TL emission after heating is contributed both by undamaged zircon as well as the reconstituted expanded zircon phase (Z₄), then the normalized TL ratio (TLᵣ) for zircon with any value of alpha activity can be represented by:

\[ TLᵣ = \frac{TLᵣ_{\text{experimental}}}{TLᵣ_{\text{maximum}}} = Z₁ + Z₄ \]

From this equation, one can calculate the fraction of the phase that is restored by heating the undamaged zircon as a function of the alpha activity. As indicated in Figure 9, the \( TLᵣ - Z₁ \) curve matches the one obtained by Pellas for expanded zircon \( Z₄ \) (Figure 10). The present investigations seem to confirm the findings of Vaz and Senftle (1970) on gem quality zircons from Ceylon.

It is thus clear that the heat treatment of zircons at 950°C for more than 20 hours restores the expanded zircon phase, produced by action of the alpha-recoil nucleus, to normal zircon \( Z₁ \). Also the TL ratio curve (Figure 8) represents the relative...
amount of damaged zircon recovered by thermal treatment. Therefore, the non-recoverable fraction should be given by the reciprocal of the normalized TL ratio (TLR) curve. This, as indicated in Figure 9, also matches the one obtained by Peillas by adding the concentrations of the phases $Z_i$ (amorphous zircon), $Z_d$ ($SiO_2$), and $Z_s$ ($ZrO_2$).

The dependence of TL emission of zircon samples with their alpha activity is clearly indicated. The enhancement of TL emission on thermal treatment of zircon samples is also alpha activity dependent and is due to the recovery of the deformed zircon fraction to normal zircon. This proves that higher alpha activity i.e., extent of metamictization is the reason for lowering of the sintering point of the zircon sands.

The results of DTA (Figure 3) show a distinct variation between the different zircons and the metamict varieties. As indicated by Frondel (1953) for the metamict zircons, the DTA curves generally show gradual but significant water loss at 100°C to 225°C and a single sharp peak at 880°C. The initial moisture loss seems almost absent for "A" and "B" zircons i.e., pink and yellow variety.

A somewhat "Not sharp" peak is noticed around 350°C. A sharp exothermic peak at 350°C is noticed for "F" zircons i.e., green variety. In general all the zircons show some indication of an exothermic change at 350°C, the minimum being shown by "B" zircon. While this could be due to combustion of organic matter or sulphide inclusions, it may also be due to some structural recrystallization, which is confirmed by the fact that this reaction is negligible for "A" and "B" zircons which have very low alpha activity. Also, previously heated samples did not show this reaction.

Minor doublets are also seen at approximately 1000°C for the metamict zircons and to a lesser extent in the "E" and "F" zircons and to a very faint extent in "A", "B", and "C" zircons. Cooling of the samples from the maximum temperature indicated no reaction whatsoever. Visual examination of the samples after DTA tests i.e., after being heated to 1300°C, showed that "D," and "E," and "F" zircons were completely fused (sintered into hard lumps); "C" zircon showed slight fusion whereas "A" and "B" zircons remained absolutely unaffected indicating thereby the stability of the samples with low alpha activity. This is further confirmed by the results of sintering tests (Table I) as the "A" and "B" zircons did not sinter at the maximum temperature of 1350°C attained in the furnace, whereas "F" zircon showed sintering at around 1250°C.

The standard 26 for crystalline zircon is 35.65. On the basis of Holland and Gottfried (1935) the decreasing 26 values for 112 for the various zircons would imply the increasing order of crystalline decay even though the variation in some cases is hardly discernible. The known metamict varieties did not yield any peak for 112.

The scanning electron micrograph of a typical grain of sample "F" zircon shown in plate 11 shows that this zircon possesses the poorest crystallinity. The micrograph indicates that fracture lines radiate from the centre of the grain, a phenomenon typical of the occurrence of metamictization.

From the present investigations unsuitability of some zircon sands, due to their higher alpha activity, for industrial use is clearly borne out.

1 Publisher's note: The magnification factor given under each electron micrograph on the following pages does not take into account the reduction necessary to fit the page size of this journal. Numbers 1-5 were reduced to 64 percent and numbers 6-11 to 75-82 percent of their original size.
Malaysian Zircon Aggregate. Very euhedral zircon grains are noticed. In addition, rutile and coarse pyrite grains can also be seen.

Indian Zircon Aggregate. Heavy ferrous coating of some grains is noticeable. Distinct grains of rutile and ilmenite are also seen. Some overgrowth is noticeable.

West Austrian Zircon Aggregate. Rounded particles of pyrites and magnetite particles are abundant with zircon.

Florida Zircon Aggregate. Rutile, pyrite, magnetite and chalcopyrite grains are seen associated with zircon.

Southport Zircon Aggregate. Pronounced colour variation with regard to internal reflection is noticed.
Scanning electron micrograph 6 X 100
Malaysian zircon showing shape habits of grains. The variation of grain shape is seen—showing predominance of crystal habits.

Scanning electron micrograph 8 X 100
West Austrian zircon showing predominance of crystal habits.

Scanning electron micrograph 7 X 100
Indian zircon showing variation of grain shape and predominance of crystal habits.

Scanning electron micrograph 9 X 200
Florida zircon showing variation of grain shape.
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